Comparative study of specific heat measurements in $LaMnO_3$, $La_{1.35}Sr_{1.65}Mn_2O_7$, $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$

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Abstract

We present the temperature dependence of the specific heat, without external magnetic field and with *9 T*, for $LaMnO_3$, $La_{1.35}Sr_{1.65}Mn_2O_7$, $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$ single crystals. We found that spin-wave excitations in the ferromagnetic and bilayer-structure $La_{1.35}Sr_{1.65}Mn_2O_7$ were suppressed by the *9 T* magnetic field. On the other hand, the external magnetic field had no effect in the specific heat of the other three antiferromagnetic samples. Also, the electronic part of the interactions were removed at very low temperatures in the $La_{1.5}Sr_{0.5}NiO_4$ single crystal, even with a zero applied magnetic field. Below *4 K*, we found that the specific heat data for $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ crystals could be fitted to an exponential decay law. Detailed magnetization measurements in this low temperature interval showed the existence of a peak close to 2 K. Both results, magnetizations and specific heat suggest the existence of an anisotropy gap in the energy spectrum of $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ compounds.

I. INTRODUCTION

The combined ordering of charge (CO) and spin (SO) is proving to be a common phenomenon in transition-metal oxides like $LaMnO_3$, $La_{1.35}Sr_{1.65}Mn_2O_7$, $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$. The magnetic properties of Mn, Co and Ni perovskites are considered to arise from the strong competition involving ferromagnetic and antiferromagnetic interactions and the spin-phonon coupling [1]− [7]. The dimensionality of the relevant structure involving the transition metal ions, three (3D) or quasi-two-dimensional (2D), also plays an important role. For instance, the bilayer-structure compounds $La_{2-2x}Sr_{1+2x}Mn_2O_7$, in which MnO_2 and $(La,Sr)_2O_2$ layers are stacked alternatively have 2D electronic and magnetic properties [8].

The well known $LaMnO_3$ is an antiferromagnetic insulator with 3D characteristics. The specific heat at low temperature, for $LaMnO_{3+\delta}$ samples, was found by L. Ghivelder et al. [9] to be very sensitive to small variations of δ. Previous specific heat measurements in the related $Nd_{0.5}Sr_{0.5}MnO_3$, $Nd_{0.5}Ca_{0.5}MnO_3$, $Sm_{0.5}Ca_{0.5}MnO_3$, $Dy_{0.5}Ca_{0.5}MnO_3$ and $Ho_{0.5}Ca_{0.5}MnO_3$ samples revealed a Schottky-like anomaly at low temperatures [10], [11]. Differently from the $LaMnO_3$ crystal, the $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$ compounds have their magnetic ions (Ni and Co) confined in planes which are insulated by $(La,Sr)_2O_2$ layers. I. A. Zaliznyak et al. [12] presented elastic and quasielastic neutron scattering measurements characterizing peculiar short-range charge-orbital and spin order in the layered perovskite $La_{1.5}Sr_{0.5}CoO_4$. They found that, below $T_c = 750\ K$, holes introduced by Sr doping lose mobility and enter into a statically ordered charge-glass-phase with loosely correlated checkerboard arrangement of empty and occupied $d_{3z^2-r^2}$ orbitals (Co^{3+} and Co^{2+}). $La_{1.5}Sr_{0.5}NiO_4$, like its parent compound La_2NiO_4, is an insulator, contrary to the related compound La_2CuO_4, where the antiferromagnetic insulator phase is rapidly destroyed by doping, leading to a metallic superconductor phase at moderate hole concentration [13], La_2NiO_4 remains nonmetallic up to quite large hole concentrations [14]. R. Kajimoto et al. [15] studied the CO in $La_{1.5}Sr_{0.5}NiO_4$ with neutron diffraction technique. They found a rearrangement of CO from checkerboard-type to stripe-type as a function of temperature. In their measurements the stripe phase persisted up to x = 0.7 for highly hole-doped samples of $Nd_{2-x}Sr_xNiO_4$ with $0.45 \leq x \leq 0.7$.

A large number of papers discuss the properties of Mn, Co and Ni perovskite compounds treated separately. However, to our knowledge, a comparative study of the low temperature specific heat for these perovskite families is missing. Here, we present the temperature dependence of the specific heat, without external magnetic field (H) and with $H=9\ T$, for $LaMnO_3$, $La_{1.35}Sr_{1.65}Mn_2O_7$, $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$ single crystals. We found that the specific heat data for $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ crystals could be fitted to an exponential decay law below $4\ K$. Detailed magnetization measurements in this low temperature interval showed the existence of a peak close to 2 K. Both results, magnetization

and specific heat suggest the existence of an anisotropy gap in the energy spectrum of $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ compounds. Our macroscopical measurements confirm the complex magnetic excitation and electronic band structure due to charge ordering and the quasi-2D confinement of the magnetic ions.

II. EXPERIMENTAL METHODS

Large single crystals of $LaMnO_3$, $La_{1.35}Sr_{1.65}Mn_2O_7$, $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$ were grown by the floating zone method described elsewhere [16]. The magnetization measurements were done with a Quantum Design MPMS-5S SQUID magnetometer. Specific heat measurements were made with a Quantum Design PPMS calorimeter that uses a *two relaxation times* technique, and data was always collected during sample cooling. The intensity of the heat pulse applied to the sample was calculated to produce a variation in the temperature bath of *0.5 %*. Experimental errors during the specific heat and magnetization measurements were lower than *1 %* for all temperatures and samples.

III. RESULTS AND DISCUSSION

Specific heat measurements at low temperatures give valuable information about the ground state excitations. In contrast to magnetization, which has a vector character, the specific heat is an scalar property. Figures 1a, 1b, 1c and 1d show the dependence of the specific heat measurements with temperature, between 2 and 30 K, for $LaMnO_3$, $La_{1.35}Sr_{1.65}Mn_2O_7$, $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$ single crystals, studied with $H=0$ and *9 T*. The data is plotted as C/T vs. T^2 to facilitate the interpretation. The $LaMnO_3$ and $La_{1.5}Sr_{0.5}CoO_4$ samples showed both an almost linear behavior and no magnetic field dependence. On the other hand, the magnetic field strongly affected the low temperature ($T < 5\ K$) behavior of the $La_{1.35}Sr_{1.65}Mn_2O_7$ compound. The specific heat curves for the $La_{1.5}Sr_{0.5}NiO_4$ sample did not change with the applied field, but were not linear at temperatures below *5 K*. Continu-

ous lines in figure 1 indicate the fitting of the experimental data between 2 and 30 K by the following expression [17]:

$$C = \sum \beta_{2n+1} T^{2n+1} + \beta_{3/2} T^{3/2} \quad (1)$$

The whole temperature interval, from *2 K* to *30 K*, was possible to be fitted with natural values of n from *0* to *4*. C is the specific heat, T is the temperature and β parameters represent the contributions of electron interactions (β_1), ferromagnetic spin waves ($\beta_{3/2}$) and phonon modes (β_3, β_5, β_7 and β_9). The coefficient β_1 (n=*0*) is also known as γ, and β_3 (n=*1*) as β. The results of the specific heat data fitting are shown in Table 1.

It is worth stressing that, differently from the $LaMnO_3$ crystal, the $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$ compounds have their magnetic ions (Ni and Co) confined in planes which are insulated by $(La,Sr)_2O_2$ layers. However, these latter two compounds show a different magnetic behavior compared to the quasi-2D $La_{1.35}Sr_{1.65}Mn_2O_7$ sample: the Mn ions order ferromagnetically, while the corresponding Ni and Co ions order antiferromagnetically. Therefore, as seen in figures 1c and 1d, there is not much contribution of the magnetic field into the measured specific heat.

¿From resistivity measurements the $LaMnO_3$, $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$ samples are electrical insulators at low temperatures, and applied magnetic fields up to 9 T are not strong enough to destroy this characteristic [12], [14], [18]. Therefore, we should not expect for the previous three crystals the linear contribution from free electrons to the specific heat. However, other kind of many-body excitations could also lead to a linear contribution [19]. On the other hand, the $La_{1.35}Sr_{1.65}Mn_2O_7$ is metallic below about 100 K [8]. Our fitting shows that β_1 values are big for $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$. To facilitate even more the comparison, we have re-plotted in figure 2 the data for the $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ crystals at temperatures below 10 K. Closed symbols represent the measurements with *H=0* and open ones with *9 T*.

Okuda et al. [8] found that the decrease of specific heat at low temperatures, due to an applied magnetic field of 9 T, was about ten times larger in a $La_{1.35}Sr_{1.65}Mn_2O_7$ sample

than the observed values in $La_{1-x}Sr_xMnO_3$ samples (with x=0.3 and 0.4). They also calculated the theoretical reduction in specific heat upon application of a magnetic field for the ideal simple-cubic (3D) and simple-square (2D) lattices and concluded that the observed change in specific heat for the bilayered manganite was large, but still less than that for an ideal 2D ferromagnetism. Besides, Okuda et al. [8] reported values of β_1=3 mJ/mole-K^2 for a $La_{1.35}Sr_{1.65}Mn_2O_7$ sample, an order of magnitude smaller than the one found by us. Because their β_1 value was similar to the one found in three dimensional perovskites, like $La_{0.7}Sr_{0.3}MnO_3$ [20], they concluded that dimensionality did not affect the value of the electron-electron interaction constant. However, our result shows that the quasi-2D confinement of the electrons in $La_{1.35}Sr_{1.65}Mn_2O_7$ do increase the electron-electron interaction constant in comparison to the 3D counterpart.

As expected, a term of the type $T^{3/2}$ appears only for the $La_{1.35}Sr_{1.65}Mn_2O_7$ sample due to its ferromagnetic interactions. B. F. Woodfield et al. [21] studied the specific heat in $La_{1-x}Sr_xMnO_3$ with x between 0.1 and 0.3 and found $\beta_{3/2}$ values in the interval 0.9 to 3.7 mJ/mole-$K^{5/2}$. Our $\beta_{3/2}$ values, for $La_{1.35}Sr_{1.65}Mn_2O_7$, are approximately equals to the 3D counterpart and roughly duplicate upon the application of a 9 T magnetic field. Figure 1c (also in fig 2) shows that the $La_{1.5}Sr_{0.5}NiO_4$ curves make a downward turn at low temperatures in both *H=0* and *9 T* and this region is not well fitted by equation 1. The values of β_1 for this sample (see table 1) apply only for temperatures higher than 4 K, and reveal a very high electronic interactions.

Martinho et al. [22] interpreted the specific heat of $La_{2-2x}Sr_{1+2x}Mn_2O_7$ samples (*0.29*$<x<$*0.51*) as thermal excitations of a two-dimensional gas of ferromagnetic magnons. However, they did not discuss the very low temperature interval (below 4 K). In figure 3 we re-scale the specific heat data in the temperature interval below *4 K* for the $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ crystals. The x-axis is now equal to the inverse of temperature and the y-axis in presented in logarithmic scale to facilitate the comparison with an exponential decay law. In both, *H=0* and *9 T*, the data points are well fitted by straight lines. The estimated energy gap (E_{gap}) in the $La_{2-2x}Sr_{1+2x}Mn_2O_7$ crystal was *0.30 meV* and *0.57 meV* for zero and 9 T,

respectively. On the other hand, the estimated E_{gap} in the $La_{1.5}Sr_{0.5}NiO_4$ crystal was *0.63 meV* and *0.65 meV* for zero and *9 T*, respectively. The graph for $La_{1.5}Sr_{0.5}NiO_4$ remind us the structure-related superconductor $La_{1.5}Sr_{0.5}CuO_4$. In a superconductor an exponential decay in the specific heat is interpreted as the opening of a gap in the electronic structure [23]. However, our sample, differently from superconductors, did not show a noticeable dependence with a magnetic field up to *9 T*. If a BCS-like theory [23] were to be valid in this crystal ($E_{gap} = 7/2\ k_B\ T_c$) the sample should have a corresponding critical temperature (T_c) at about *2 K*.

Figure 4 shows a detailed measurement of the zero field cooling (*ZFC*) magnetization in the $La_{1.35}Sr_{1.65}Mn_2O_7$ crystal, between 1.8 and 10 K, with applied magnetic field of 20 Oe (a) and 50 Oe (b). Measurements were done with the magnetic field parallel (triangles) and perpendicular (squares) to the *c* axes and temperature steps of *0.1 K*. The results reveal a clear anisotropy due to the quasi-bidimensional distribution of the magnetic ions. The magnetization shows three features: a minimum close to 2 K, a small maximum close to 4 K and a plateau close to 7 K. Figure 5 shows *ZFC* magnetization measurement in the $La_{1.5}Sr_{0.5}NiO_4$ crystal between 1.8 and 10 K with applied magnetic field of 1 T (a) and 3 T (b). The magnetic field was applied parallel (triangles) and perpendicular (squares) to the *c* axes and the temperature steps were of *0.1 K*. These graphs also display an anisotropy behavior due to the quasi-bidimensional distribution of the magnetic ions. The magnetization here shows two features: a maximum close to 2 K and a minimum close to 4 K in the orientation of the applied magnetic field parallel to the *c* axes. The absolute value of magnetization is higher in $La_{1.35}Sr_{1.65}Mn_2O_7$, due to its ferromagnetic alignment, in comparison with the antiferromagnetic alignment in $La_{1.5}Sr_{0.5}NiO_4$. In both cases the position of the peaks close to 2 K in the magnetization curves (figures 4 and 5) seems to be correlated to the exponential decay in the specific heat (figure 3).

Recently, Boothroyd [24] et al. studied a single crystal of $La_{1.5}Sr_{0.5}NiO_4$ with polarized neutrons at 10 K. They made neutron energy scans with the direction of the incident beam fixed and aligned parallel, as well as perpendicular, to the NiO layers. They found inter-plane

correlations at low energies (negligible for E $\geq$ 5 meV) and a reduction in intensity below an energy of 4 meV. Given that neutrons scatter from spin fluctuations perpendicular to the wave vector, these observations indicated that the intensity reduction below 4 meV was due to the freezing out of the c-axis component of the spin fluctuations. They also pointed out the existence of a 4 meV energy gap due to single-ion out-of-plane anisotropy. Although we found in the $La_{1.5}Sr_{0.5}NiO_4$ crystal, using specific heat measurements, a gap value smaller than the one reported by Boothroyd et al. [24], both results, one microscopically and the other macroscopically, seem to confirm the complex magnetic excitation and band structure due to charge ordering and the quasi-2D confinement of the Ni ions. Further studies are clearly necessary to elucidate better these points.

IV. CONCLUSIONS

Single crystals of $LaMnO_3$, $La_{1.35}Sr_{1.65}Mn_2O_7$, $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$ were characterized by magnetization and specific heat measurements. The bilayer compound $La_{1.35}Sr_{1.65}Mn_2O_7$ presents a ferromagnetic transition, while the other studied compositions show an antiferromagnetic order. Spin-wave excitations in the bilayer-structure $La_{1.35}Sr_{1.65}Mn_2O_7$ are suppressed by a 9 T magnetic field as indicated by specific heat measurements. This is attributed to the reduced magnetic dimensionality. The effect is larger than in the case of 3D compounds, but not as large as expected in an ideal 2D system. Unlike a previous report [8], we found that the quasi-2D confinement of the electrons in $La_{1.35}Sr_{1.65}Mn_2O_7$ do increase the electron-electron interaction constant in comparison to its 3D counterpart. The specific heat does not change with a 9 T magnetic field in $LaMnO_3$, $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$. However, electronic excitations are drastically removed at very low temperatures in the $La_{1.5}Sr_{0.5}NiO_4$ single crystal, as revealed by the downward turn in the specific heat. Below *4 K*, we also found that the specific heat data for the $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ crystals could be fitted by an exponential decay law. From the fittings we were able to estimate characteristic energy gaps for both compounds.

Detailed magnetization measurements in this low temperature interval showed the existence, close to 2 K, of a maximum for $La_{1.5}Sr_{0.5}NiO_4$ and a minimum for $La_{1.35}Sr_{1.65}Mn_2O_7$. Our measurements of magnetizations and specific heat, combined with a previous report on neutron diffraction [24], suggest the existence of an anisotropy gap in the energy spectrum of the $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ compounds.

V. ACKNOWLEDGMENTS

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Table 1. Results of the fitting to the law $C = \sum \beta_{2n+1} T^{2n+1} + \beta_{3/2} T^{3/2}$, with n from 0 to 4, for the four studied single crystals and *H= 0* and *9 T*. The units are mJ/mole-K^j, where j is the subscript of the coefficient.

Sample	H (T)	β_1	$\beta_{3/2}$	β_3	β_5	β_7	β_9
$LaMnO_3$	0	1.1	0	0.20	0.31	-0.48	2.0
	9	1.1	0	0.20	0.31	-0.48	2.0
$La_{1.35}Sr_{1.65}Mn_2O_7$	0	24	2.4	0.12	1.3	-1.4	5.3
	9	14	4.4	0.20	0.98	-1.0	3.5
$La_{1.5}Sr_{0.5}NiO_4$	0	16	0	0.18	0.67	-0.79	2.8
	9	16	0	0.17	0.71	-0.82	2.9
$La_{1.5}Sr_{0.5}CoO_4$	0	1.4	0	0.12	0.61	-0.76	2.9
	9	1.9	0	0.12	0.61	-0.76	2.9

Figure Captions

Figure 1. Specific heat measurements between 2 and 30 K in the single crystals $LaMnO_3$, $La_{1.35}Sr_{1.65}Mn_2O_7$, $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.5}Sr_{0.5}CoO_4$, with *H=0* and *9 T*. Continuous lines represent the fitting of the law: $C = \sum \beta_{2n+1} T^{2n+1} + \beta_{3/2} T^{3/2}$. The data is plotted as C/T vs. T^2 to facilitate the interpretation.

Figure 2. Specific heat of $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ crystals below 10 K. Close symbols represent the measurements with *H=0* and open ones with *9 T*.

Figure 3. Re-scaled specific heat data in the low temperature interval (below *4 K*) for the $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ crystals. The x-axis is equal to the inverse of temperature and the y-axis in presented in logarithmic scale to facilitate the test of an exponential decay law.

Figure 4. Magnetization measurements in the $La_{1.35}Sr_{1.65}Mn_2O_7$ crystal between 1.8 and 10 K with applied magnetic field of 20 Oe (a) and 50 Oe (b). Measurements were done with the magnetic field parallel (triangles) and perpendicular (squares) to the *c* axes. The results reveal an anisotropy due to the quasi-bidimensional distribution of the magnetic ions.

Figure 5. Magnetization measurements in the $La_{1.5}Sr_{0.5}NiO_4$ crystal between 1.8 and 10 K with applied magnetic field of 1 T (a) and 3 T (b). Magnetic field was applied parallel (triangles) and perpendicular (squares) to the *c* axes. The small peak close to 2 K, in the parallel orientation to the *c* axes, might be correlated with a gap in the electronic structure.

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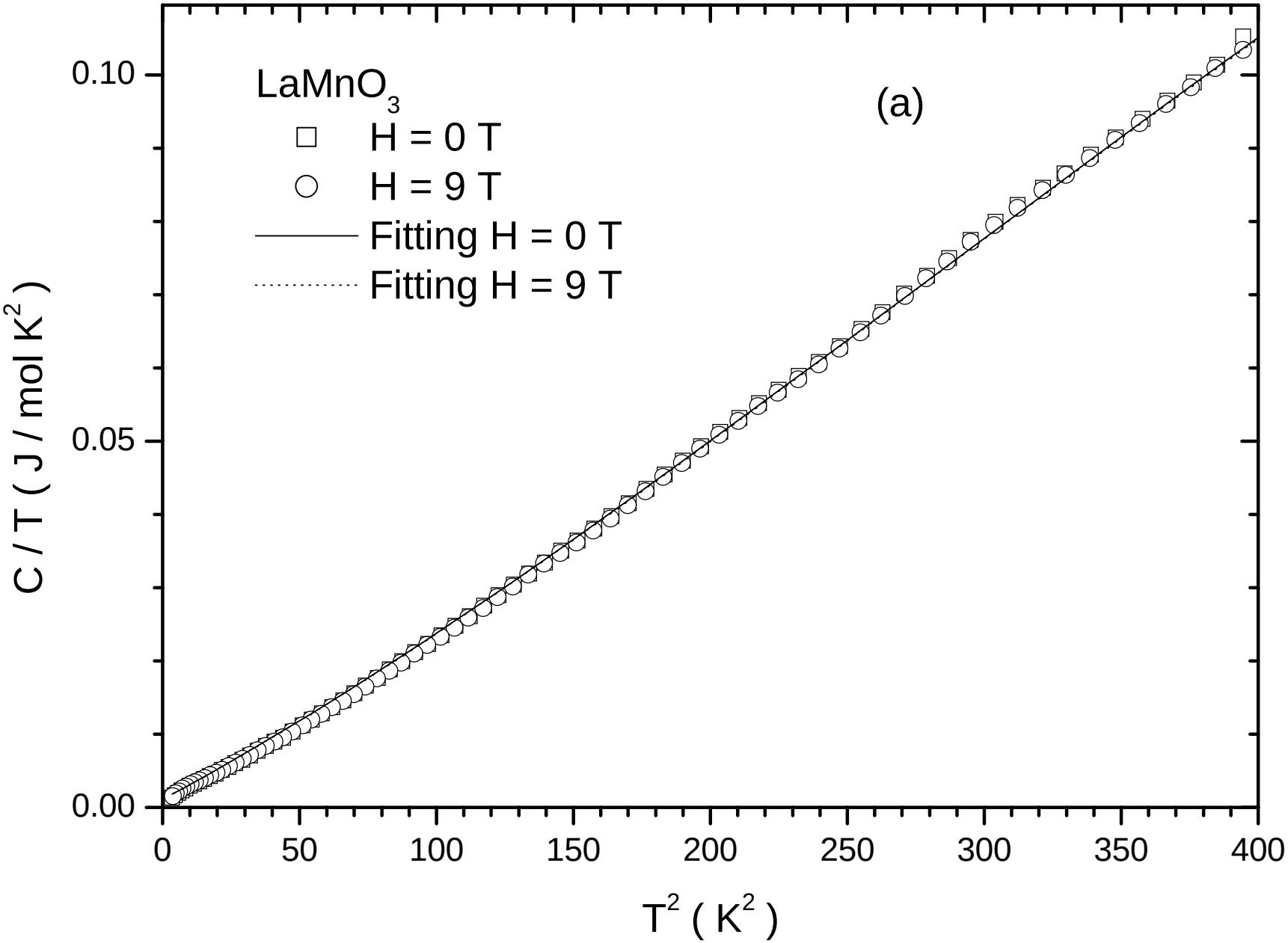
Figure 1


LaMnO3
(a)
H = 0 T
H = 9 T
Fitting H = 0 T
Fitting H = 9 T
C / T (J / mol K2)
T2 (K2)
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0.05
0.10
0
50
100
150
200
250
300
350
400

Figure 1

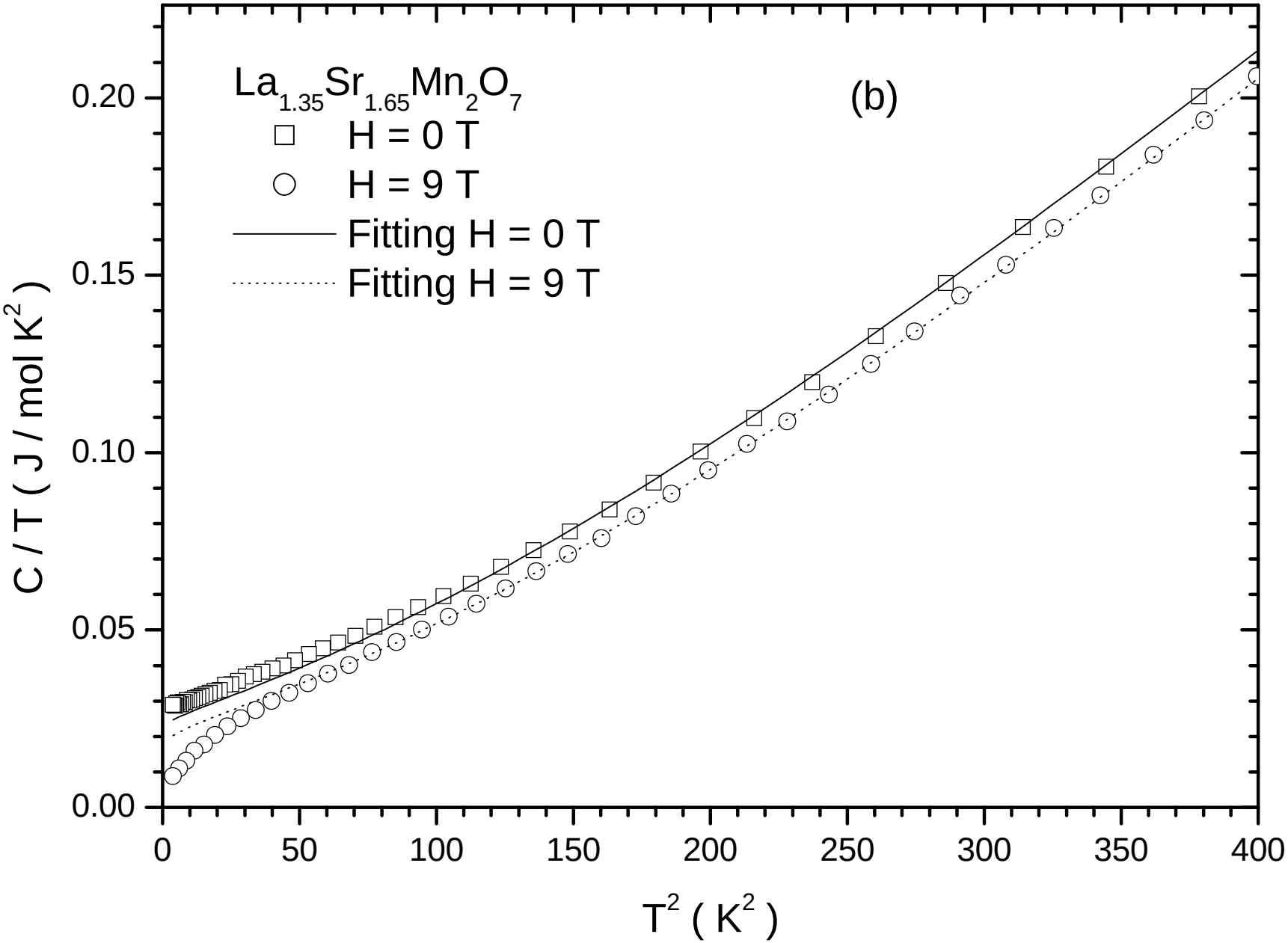

La1.35Sr1.65Mn2O7
(b)
H = 0 T
H = 9 T
Fitting H = 0 T
Fitting H = 9 T
C / T (J / mol K2)
T2 (K2)
0.00
0.05
0.10
0.15
0.20
0
50
100
150
200
250
300
350
400

Figure 1

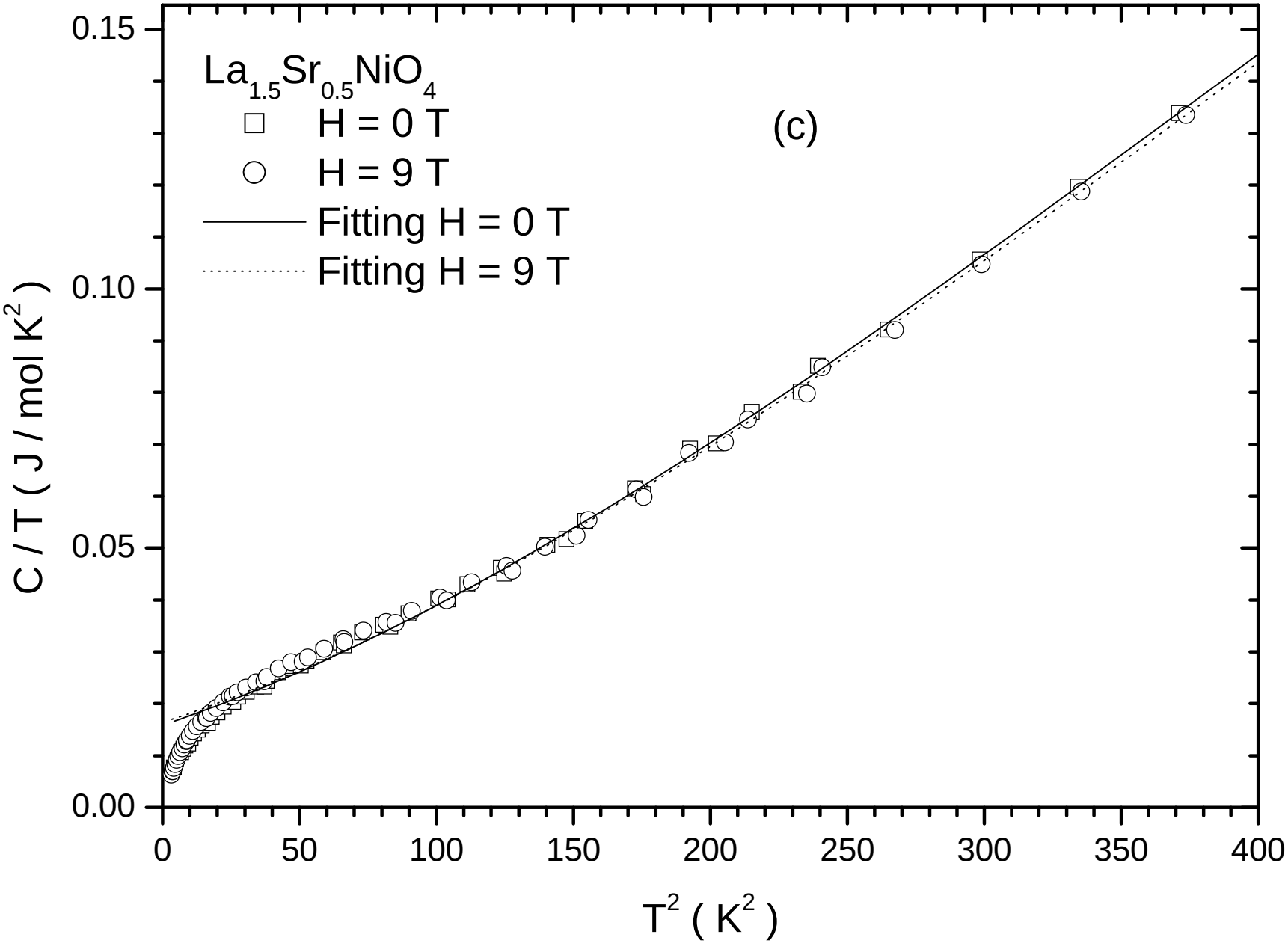

La1.5Sr0.5NiO4
H = 0 T
H = 9 T
Fitting H = 0 T
Fitting H = 9 T
(c)
C / T (J / mol K2)
T2 (K2)
0.00
0.05
0.10
0.15
0
50
100
150
200
250
300
350
400

Figure 1

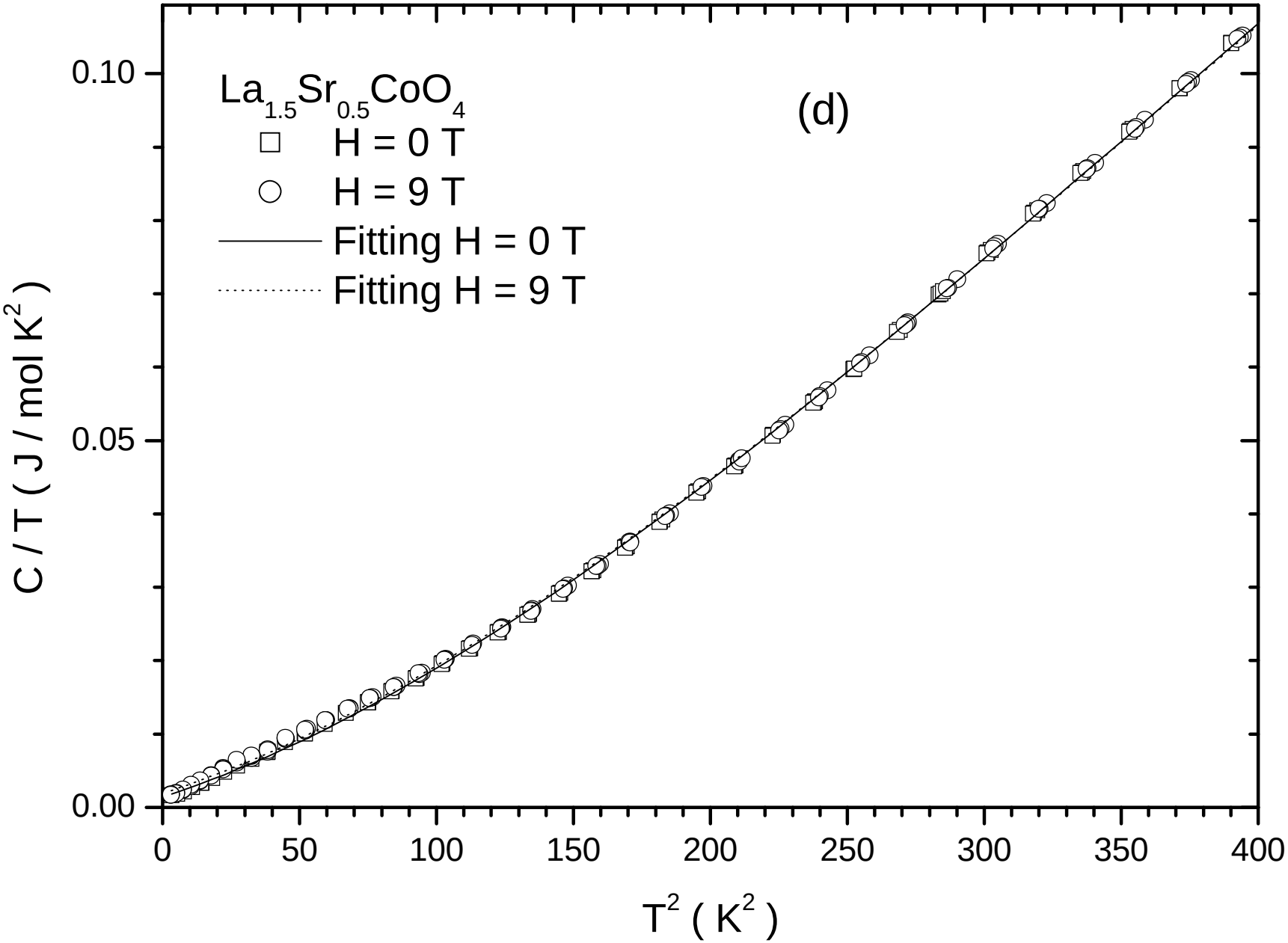

La1.5Sr0.5CoO4
(d)
H = 0 T
H = 9 T
Fitting H = 0 T
Fitting H = 9 T
C / T (J / mol K2)
T2 (K2)
0.00
0.05
0.10
0
50
100
150
200
250
300
350
400

Figure 2

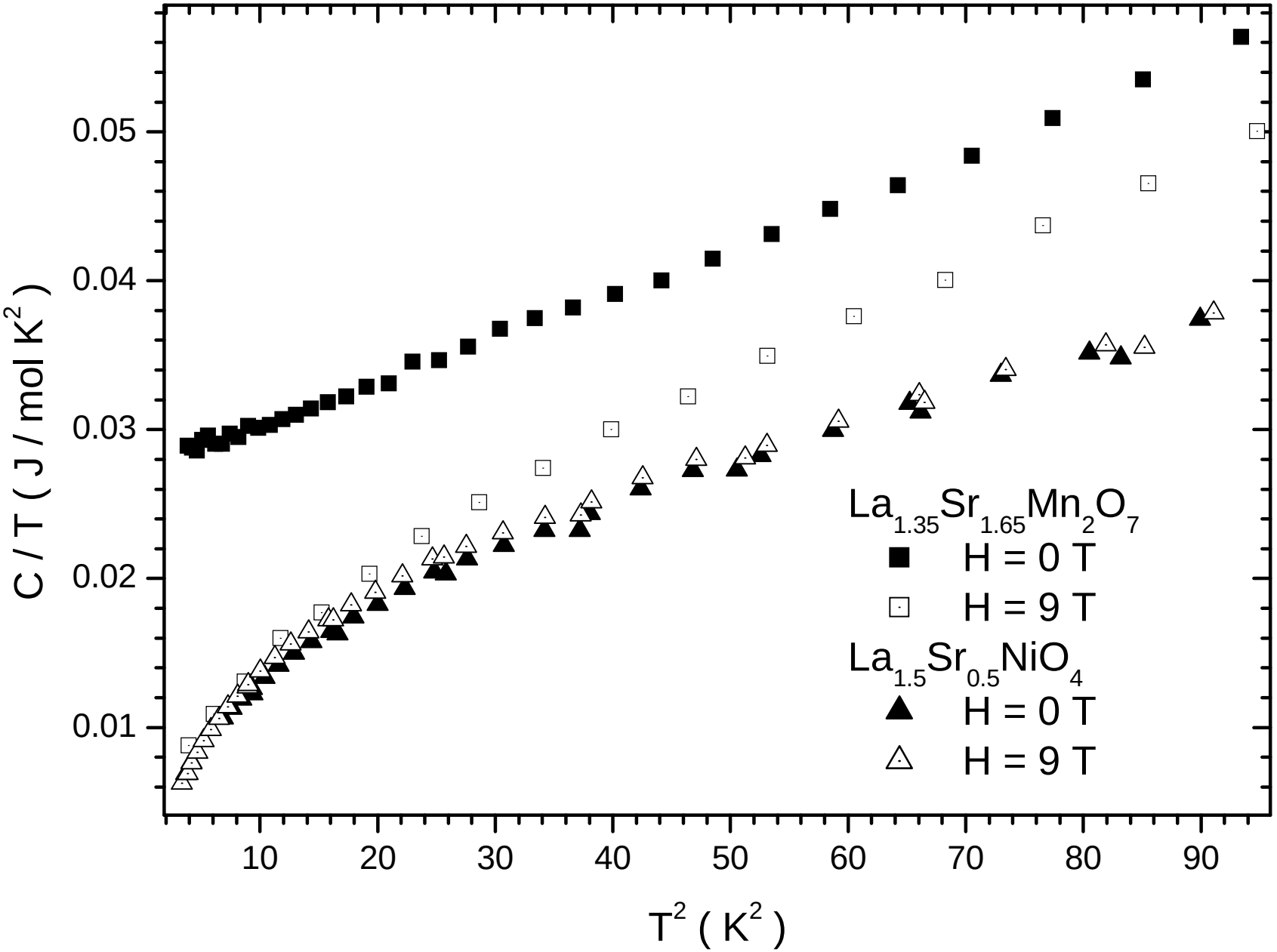

0.05
0.04
0.03
0.02
0.01
C / T (J / mol K^2)
10
20
30
40
50
60
70
80
90
T^2 (K^2)
La$_{1.35}$Sr$_{1.65}$Mn$_2$O$_7$
H = 0 T
H = 9 T
La$_{1.5}$Sr$_{0.5}$NiO$_4$
H = 0 T
H = 9 T

Figure 3

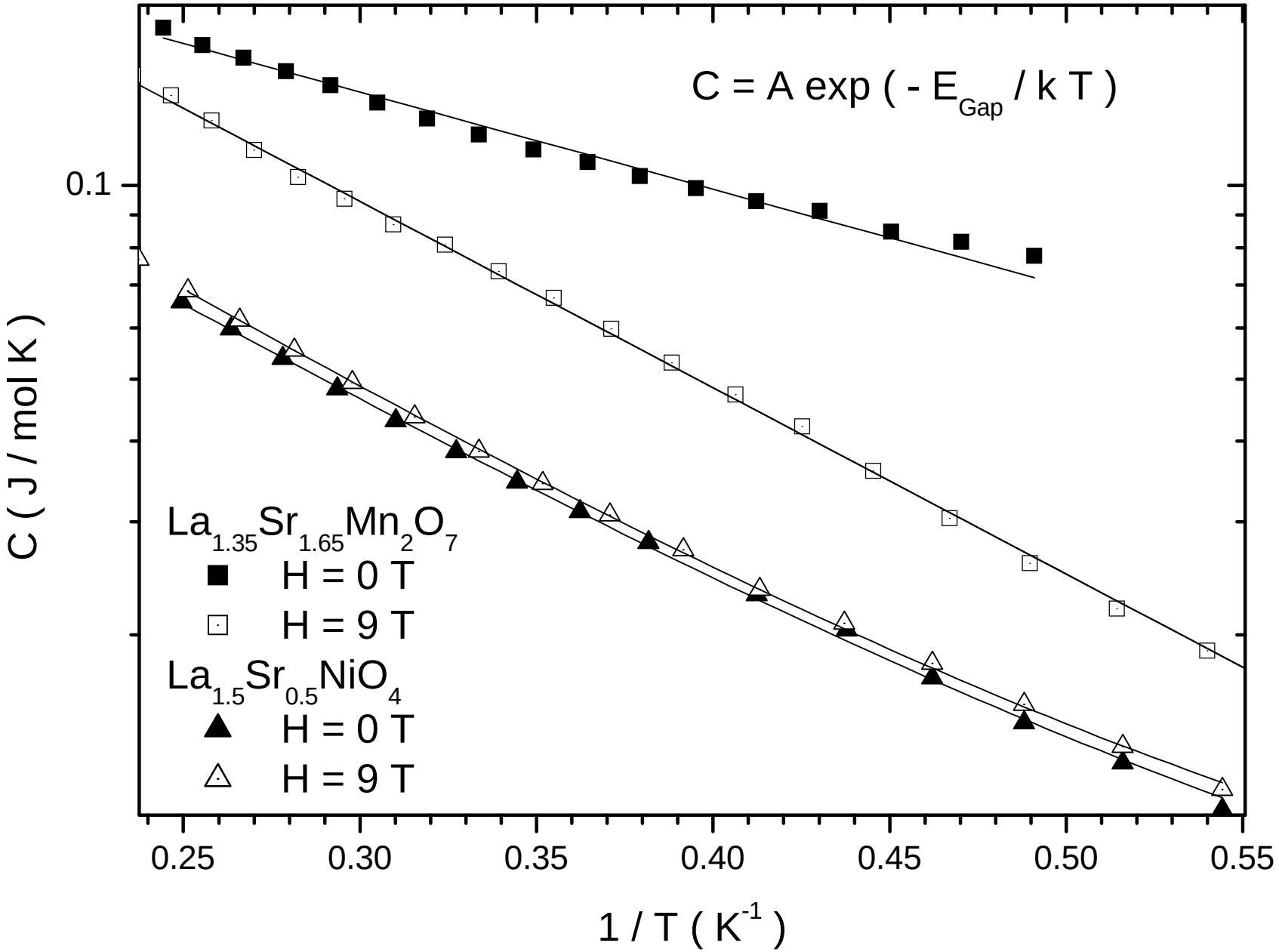

C = A exp (- E_Gap / k T)
0.1
C (J / mol K)
La1.35Sr1.65Mn2O7
H = 0 T
H = 9 T
La1.5Sr0.5NiO4
H = 0 T
H = 9 T
0.25
0.30
0.35
0.40
0.45
0.50
0.55
1 / T (K-1)

Figure 4a

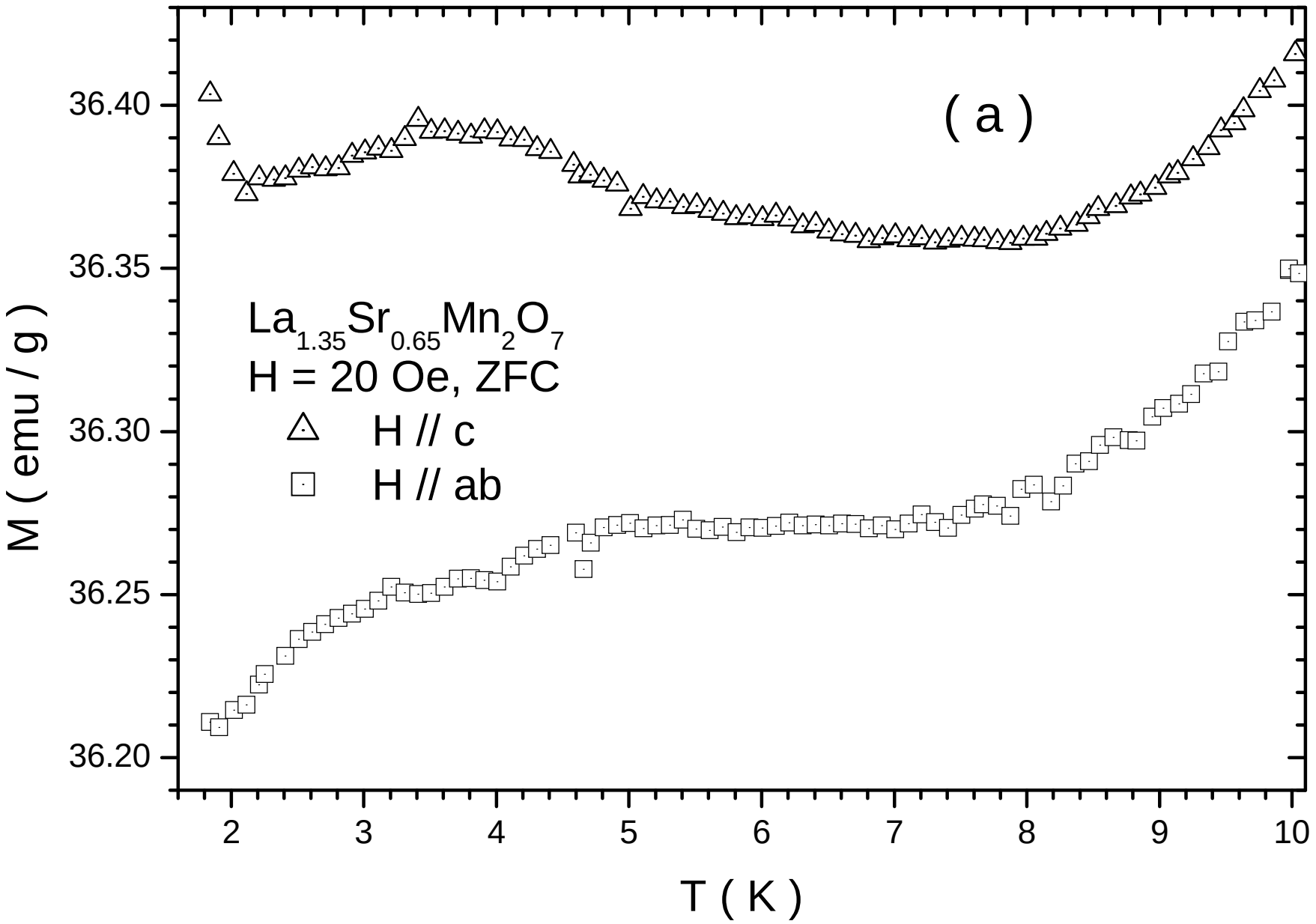

(a)
$La_{1.35}Sr_{0.65}Mn_2O_7$
H = 20 Oe, ZFC
H // c
H // ab
M (emu / g)
T (K)
36.40
36.35
36.30
36.25
36.20
2
3
4
5
6
7
8
9
10

Figure 4b

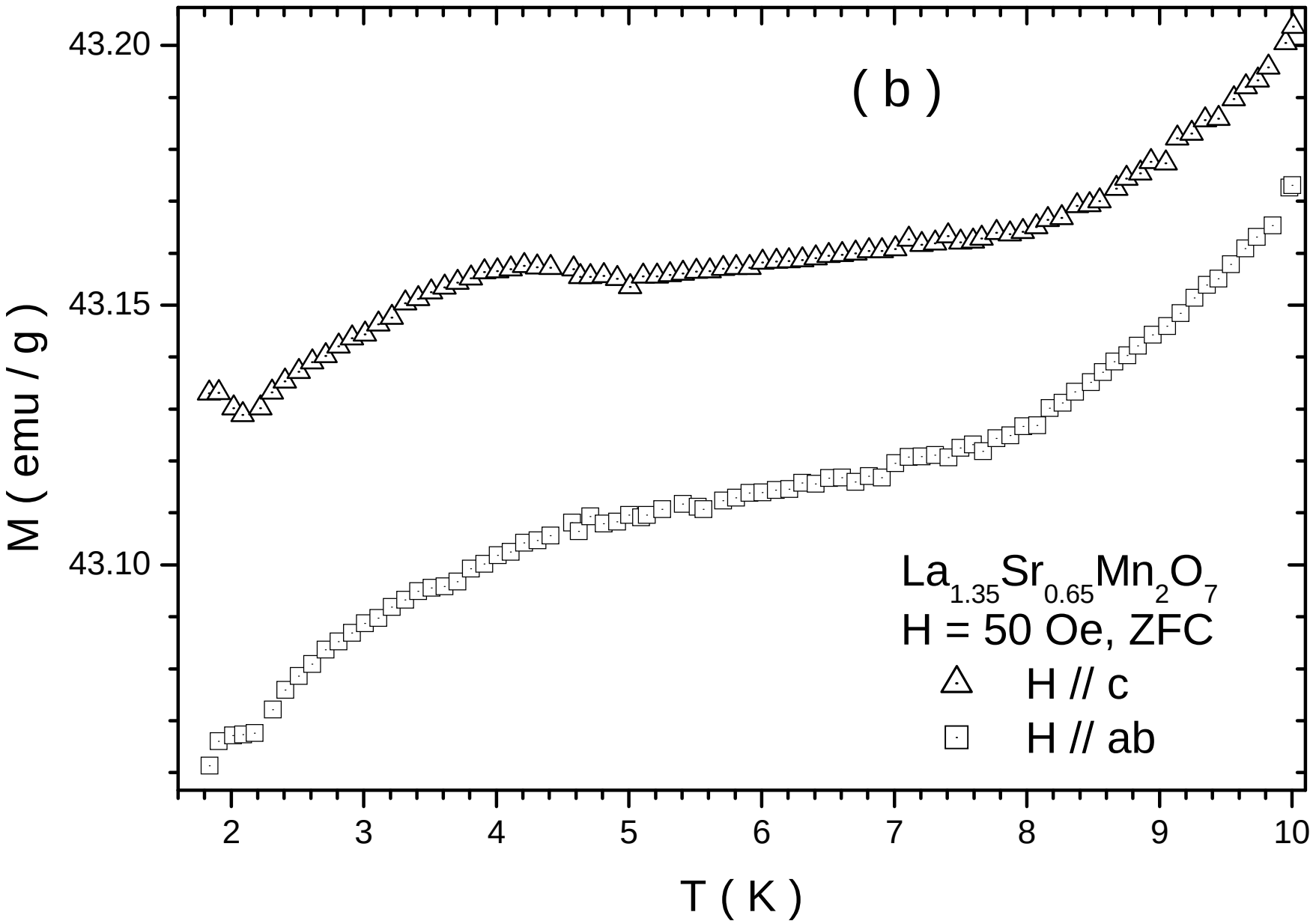

(b)
43.20
43.15
43.10
M (emu / g)
2
3
4
5
6
7
8
9
10
T (K)
La1.35Sr0.65Mn2O7
H = 50 Oe, ZFC
H // c
H // ab

Figure 5a

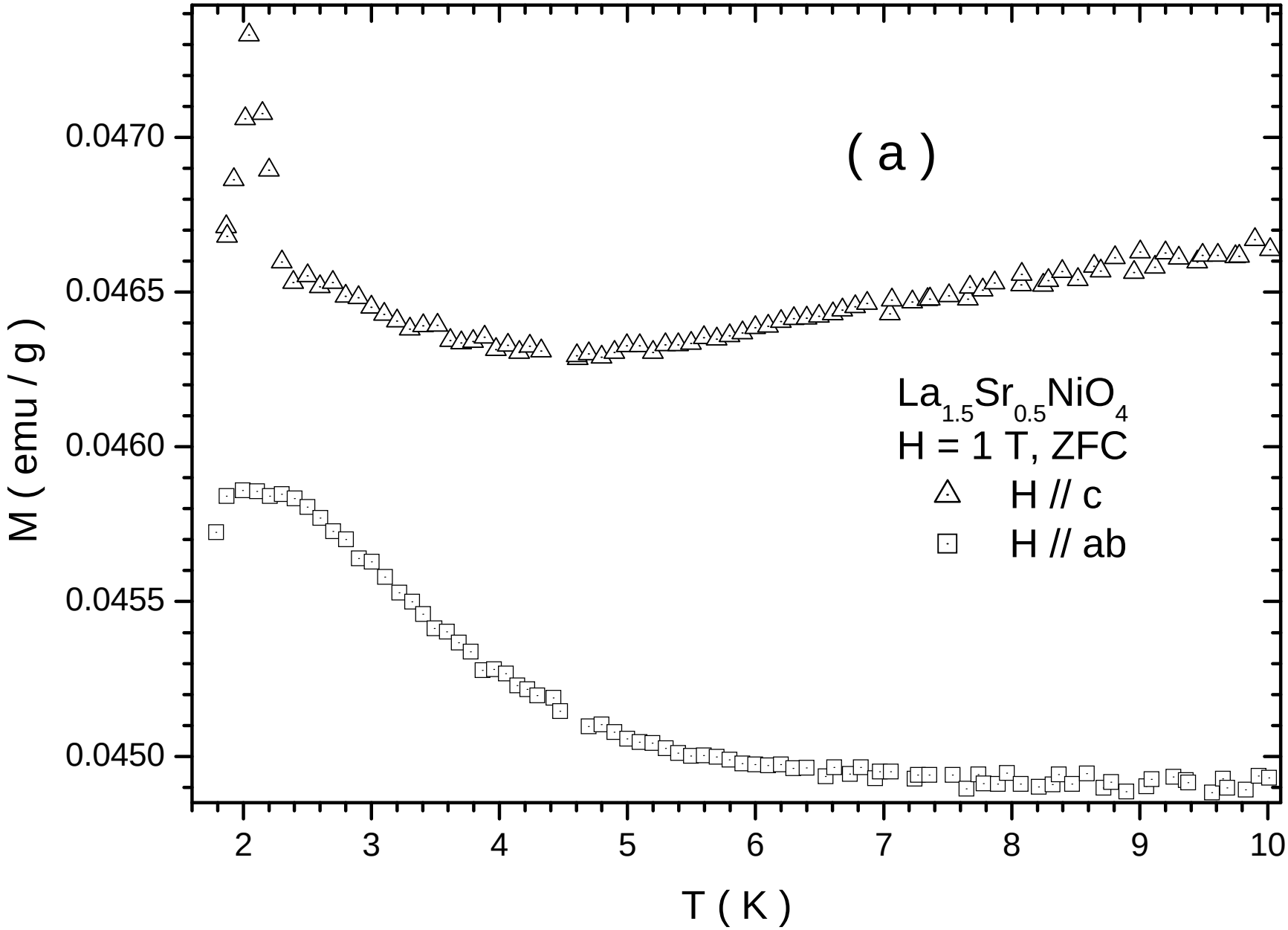
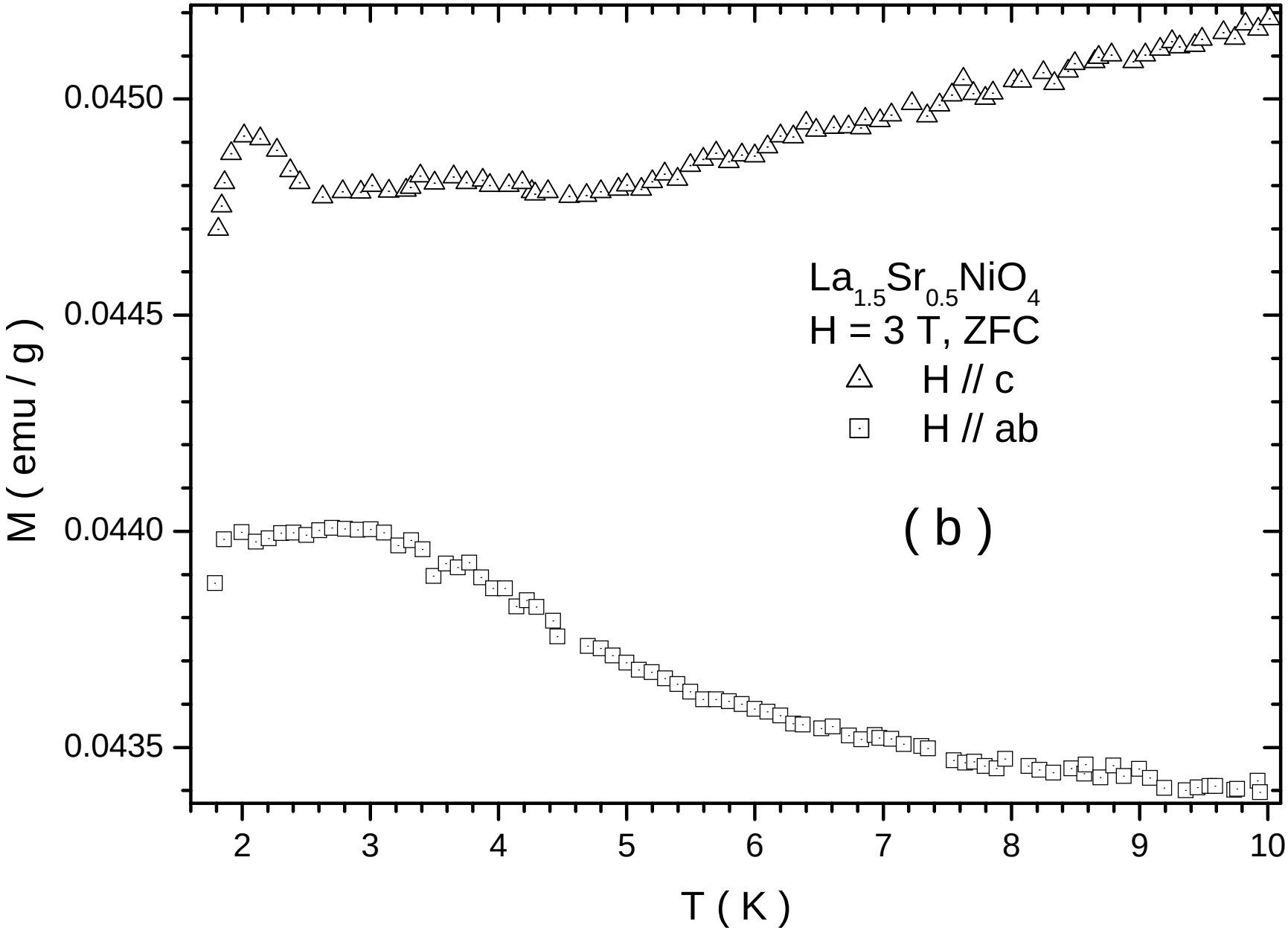

(a)
La1.5Sr0.5NiO4
H = 1 T, ZFC
H // c
H // ab
M (emu / g)
T (K)
0.0470
0.0465
0.0460
0.0455
0.0450
2
3
4
5
6
7
8
9
10

Figure 5b


0.0450
0.0445
0.0440
0.0435
M (emu / g)
2
3
4
5
6
7
8
9
10
T (K)
La1.5Sr0.5NiO4
H = 3 T, ZFC
H // c
H // ab
(b)